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ZINC(II) ARENE TELLUROLATO COMPLEXES AS PRECURSORS TO ZINC TELLURIDE. THE CRYSTAL AND MOLECULAR STRUCTURE OF $[Zn(TeC_6H_2Me_3\text{-}2,4,6)_2(pyridine)_2]$

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Abstract—The zinc tellurolato complex $[Zn(TeC_6H_2Me_3\text{-}2,4,6)_2]_n$ (**1**) was obtained by protolysis of $Zn[N(SiMe_3)_2]_2$ with mesitylene tellurol, 2,4,6-$Me_3C_6H_2TeH$, in light petroleum. The complex formed a coordination polymer soluble only in strongly coordinating solvents. Reaction with one equivalent of PMe_3 afforded $[Zn(TeC_6H_2Me_3)_2(PMe_3)]$ (**2**), while an excess of Lewis bases L gave four-coordinate adducts $[Zn(TeC_6H_2Me_3)_2L_2]$ [L = PMe_3 (**3**), *N*-methylimidazole (**5**), pyridine (**6**); $L_2 = Me_2PC_2H_4PMe_2$ (**4**)]. The adducts are more soluble than **1** but lose L on heating. The crystal structure of **6** has been determined by X-ray diffraction and confirmed the monomeric nature of the complex. Heating the complexes either without a solvent or in paraffin oil to 270–320°C led to the formation of cubic ZnTe. Prolonged exposure to temperatures of 350°C *in vacuo* led to the slow decomposition of ZnTe into the elements. The thermolysis of **1** in refluxing 4-ethylpyridine proceeded more slowly giving nano-scale particles of ZnTe, accompanied by the formation of crystalline tellurium.

Metal chalcogenolato complexes have attracted attention recently as single-source precursors to Group 12 metal chalcogenide (II–VI) semiconductor materials. In early work Takahashi *et al.*[1] and Osakada and Yamamoto[2] used metal dimethyldithiophosphinato and methylthiolato complexes, respectively, to generate polycrystalline ZnS and CdS. Steigerwald *et al.*[3] prepared nanostructure particles of ZnS, ZnSe, CdS, CdSe, CdTe and HgTe from the corresponding benzene chalcogenolato complexes and their phosphine adducts. More recently we have shown that sterically highly hindered arene chalcogenolato complexes are suitable for the deposition of II–VI films by low-pressure metal–organic chemical vapour deposition (MOCVD) techniques[4] and have reported the synthesis of arene tellurolato complexes of cadmium[5] and mercury.[6] By contrast, tellurolato complexes of zinc are rare. Arnold *et al.*[7] synthesized the silyltellurolato complex $Zn[TeSi(SiMe_3)_3]_2$ which possesses sufficient thermal stability and volatility for the vapour-phase deposition of thin ($\leqslant 500$ Å) films of ZnTe. We report here the synthesis and characterization of zinc(II) mesitylene tellurolato complexes and some Lewis base adducts and the use of these compounds as precursors to zinc telluride.[8]

RESULTS AND DISCUSSION

The protolysis of $Zn[N(SiMe_3)_2]_2$ with mesitylene tellurol, 2,4,6-$Me_3C_6H_2TeH$ (R^1TeH), in light petroleum at -78°C to room temperature led to the precipitation of $[Zn(TeR^1)_2]_\infty$ (**1**) as an off-white to pale-pink solid [eq. (1)]. The compound was poorly soluble in hydrocarbons but dissolved readily in more strongly coordinating solvents such as DMSO, DMF or pyridine. Solid **1** forms, most probably, one-dimensional infinite chains, similar to $[Cd(TeR^1)_2]_\infty$.[5] This assumption is supported by

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the IR spectrum which shows a low frequency ν(Zn—Te) band at 178 cm^{-1}, 20–40 cm^{-1} below the values found for monomeric complexes $[Zn(TeR^1)_2L_2]$. As expected for a coordination polymer, **1** is not volatile.

$$Zn[N(SiMe_3)_2]_2 + 2R^1TeH \longrightarrow \underset{\mathbf{1}}{[Zn(TeR^1)_2]} + 2HN(SiMe_3)_2 \quad (1)$$

Adding one equivalent of trimethylphosphine to a suspension of **1** in petroleum resulted in a clear solution from which a white product quickly precipitated. The product had the composition $[Zn(TeR^1)_2(PMe_3)]$ (**2**). The ν(Zn—Te) IR bands were found at 196 and 215 cm^{-1}, indicative of terminal Zn—Te bonds, and suggest that **2** may be monomeric rather than a Te-bridged dimer.[9] Molecular weight determinations in benzene were inconclusive since PMe_3 partly dissociates under these conditions. The addition of two equivalents of PMe_3 or of $Me_2PC_2H_4PMe_2$ (dmpe) to **1** afforded the expected bis(phosphine) complexes $[Zn(TeR^1)_2(PMe_3)_2]$ (**3**) and $[Zn(TeR^1)_2(dmpe)]$ (**4**) as colourless solids. The compounds are slightly soluble in dichloromethane or chloroform but insoluble in toluene. The analogous reaction of **1** with nitrogen ligands gave $[Zn(TeR^1)_2L_2]$ [L = *N*-methylimidazole (**5**) and pyridine (**6**)] [eq. (2)]. The formation of a stable pyridine adduct **6** is a reflection of the increased Lewis acidity of zinc; neither cadmium nor mercury arene tellurolato complexes form stable pyridine complexes. The adducts **2**–**6** lose the donor ligands on heating.

$$1 + 2L \longrightarrow \underset{\mathbf{3\text{–}6}}{[Zn(TeR^1)_2L_2]} \quad (2)$$

Structure of $[Zn(TeR^1)_2(py)_2]$ (**6**)

The structure of the pyridine complex **6** was determined by X-ray diffraction. A view of the molecule is shown in Fig. 1. Important bond lengths and angles are collected in Table 1. The complex possesses a distorted tetrahedral geometry. Unlike other three- and four-coordinate arene chalcogenolato complexes of zinc[9] the two tellurolato ligands in **6** adopt a conformation with co-planar mesityl groups. The steric repulsion between these bulky substituents is reflected in the wide Te—Zn—Te angle of 126.9(1)°, while the N—Zn—N angle is only 89.2(3)°. The Zn—Te bond distance of 2.573(1) Å is almost identical to the Zn—Te distance in the related silyltellurolato complexes $[Zn\{TeSi(SiMe_3)_3\}_2(py)_2]$.[10]

Thermolysis studies

The thermolysis of **1**, **2**, **3** and **5** in the solid state *in vacuo* led to the formation of microcrystalline ZnTe, as monitored by X-ray powder diffraction.[11] The results are shown in Table 2. Heating **1** to 320°C for 4 h, followed by another 4 h at 350°C gave mainly cubic ZnTe. Some decomposition into metallic zinc and tellurium is also apparent. If adducts such as **2**, **3** and **5** are used, the thermolysis starts with the dissociation of the donor ligands. The main organic by-product is R^1_2Te [eq. (3)].

$$Zn(TeR^1)_2L_2 \longrightarrow ZnTe + R^1_2Te + 2L. \quad (3)$$

When a solution of **1** in 4-ethylpyridine was heated to reflux, the colour changed slowly from pale yellow to dark brown. After 20 min a new absorption band at 490 nm appeared in the UV–vis spectrum which was replaced by an absorption at 550 nm on prolonged heating. After 6 h only the band at 550 nm was observed, and a dark brown precipitate formed. Samples for powder X-ray diffraction after heating for 10 h showed a rather broad diffraction pattern of partially crystalline cubic ZnTe, together with traces of tellurium. A similar pattern was obtained from samples of **1** heated in paraffin oil at 270°C for 6 h (Fig. 2a). Annealing this product under argon in a quartz tube at 700°C gave ZnTe with a well-defined diffraction pattern (Fig. 2b). Since ZnTe is unstable at these temperatures, the annealing process is accompanied by the deposition of some elemental tellurium and zinc in the cooler parts of the tube.

A heated solution of **2** in 4-ethylpyridine showed absorptions at 410, 480 and 560 nm after 15 min, 2 h and 24 h, respectively, indicative of the build-up of increasingly large clusters. However, there was no evidence for the formation of solid products at this stage, and attempts to isolate particles from these 4-ethylpyridine solutions using an ultracentrifuge failed. After 90 h heating a brown precipitate appeared, and the 560 nm band disappeared. The comparatively slow particle growth of mononuclear precursors such as **2** in a strongly coordinating solvent suggests that the process may be suitable for the synthesis of particles of pre-determined size.

CONCLUSION

Bis(mesitylene tellurolato)zinc is accessible by the protolysis of $Zn[N(SiMe_3)_2]_2$ with $C_6H_2Me_3$-TeH. The complex is polymeric but forms monomeric Lewis base adducts. Polymeric and monomeric complexes are insufficiently volatile for gas-phase film depositions but decompose in the solid

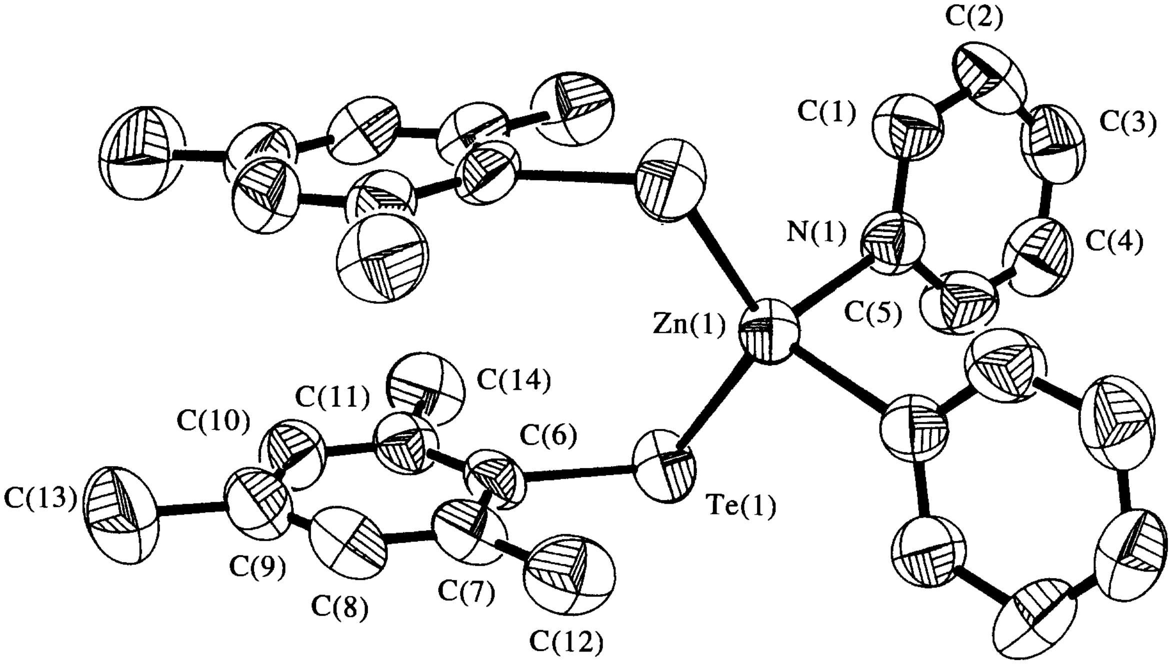

Fig. 1. Molecular structure of $[Zn(TeR^1)_2(py)_2]$ (**6**), showing the atomic numbering scheme. Ellipsoids are drawn at 30% probability.

Table 1. Selected bond lengths (Å) and angles (°) of $[Zn(TeR^1)_2(py)_2]$ (**6**)[a]

Te(1)—Zn(1)	2.573(1)	Te(1)—C(6)	2.140(6)
Zn(1)—N(1)	2.110(5)	N(1)—C(1)	1.342(7)
N(1)—C(5)	1.346(10)	C(1)—C(2)	1.392(11)
C(2)—C(3)	1.344(12)	C(3)—C(4)	1.369(11)
C(4)—C(5)	1.363(11)		
Zn(1)—Te(1)—C(6)	101.1(2)	Te(1)—Zn(1)—N(1)	106.8(2)
Te(1)—Zn(1)—Te(1A)	126.9(1)	N(1)—Zn(1)—Te(1A)	110.4(1)
N(1)—Zn(1)—N(1A)	89.2(3)	Zn(1)—N(1)—C(1)	122.8(5)
Zn(1)—N(1)—C(5)	120.2(4)	Te(1)—C(6)—C(7)	121.5(5)
Te(1)—C(6)—C(11)	118.6(5)		

[a] The atoms Te(1) and Te(1A) and N(1) and N(1A) are related by rotation about the C_2 axis.

Table 2. Thermolysis of zinc tellurolato complexes

Compound	Solvent	Oven temperature	Time	Solid product
$Zn(TeR^1)_2$ (**1**)	—	320°C	4 h	cubic ZnTe, small amounts of
		350°C	4 h	Zn and Te
$Zn(TeR^1)_2(PMe_3)_2$ (**3**)	—	320°C	2 h	cubic ZnTe
$Zn(TeR^1)_2(dmpe)$ (**4**)	—	320°C	8 h	cubic ZnTe
$Zn(TeR^1)_2(imid)_2$ (**5**)	—	320°C	8 h	cubic ZnTe
$Zn(TeR^1)_2$ (**1**)	Paraffin oil	270°C	6 h	cubic ZnTe (poorly crystalline), minor amounts of Te
$Zn(TeR^1)_2$ (**1**)	4-Etpy	168°C	10 h	Te, poorly crystalline ZnTe

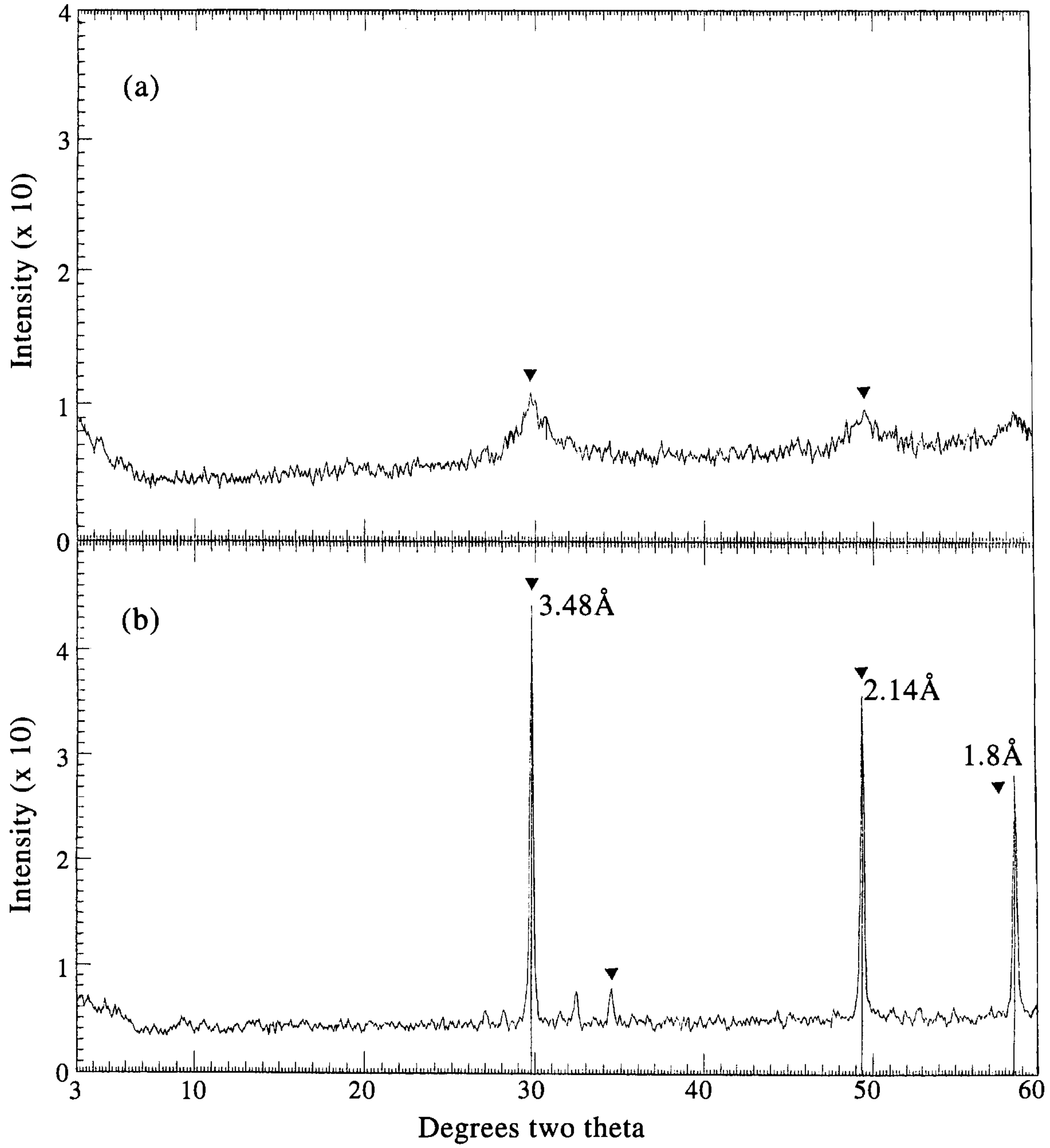

Fig. 2. X-ray pattern of the thermolysis product of **1** in paraffin oil; (a) as prepared at 270°C, (b) after annealing at 700°C, showing the diffraction pattern of cubic ZnTe. ▼ denotes reflections assigned to cubic ZnTe.

state to give polycrystalline ZnTe. Heating solutions in coordinating solvents such as 4-ethylpyridine leads to slow growth of colloidal ZnTe particles, while thermolysis in non-coordinating media such as paraffin oil generates finely divided ZnTe suspensions.

EXPERIMENTAL

General procedures

All reactions were carried out under inert gas using standard vacuum-line techniques. Solvents were distilled under nitrogen from sodium benzophenone [diethyl ether, thf, petroleum ether (bp. 40–60°C)], sodium (toluene) or calcium hydride (dichloromethane). The NMR solvents were stored over 4Å molecular sieves under nitrogen or argon and degassed by several freeze–thaw cycles. The compounds 2,4,6-Me$_3$C$_6$H$_2$TeH (R^{1}TeH)[5] and Zn[N(SiMe$_3$)$_2$]$_2$[12] were prepared as previously described. PMe$_3$, Me$_2$PC$_2$H$_4$PMe$_2$ and *N*-methylimidazole (imid) were used as purchased. Melting points are uncorrected. Infrared spectra in the 600–50 cm^{-1} region were recorded on a Mattson Polaris spectrometer and NMR spectra on a JEOL EX270 instrument. Powder X-ray measurements were carried out using a Phillips PW1710 diffractometer using Co-K_α radiation (λ = 1.790260 Å).

Synthesis of Zn(TeR^1)$_2$ (**1**)

To a solution of 4.4 mmol R^{1}TeH, prepared *in situ* from 1.086 g (2.20 mmol) R$^1{}_2$Te$_2$ as described,[5] in 50 cm^3 petroleum ether was added 0.85 g (2.20 mmol) Zn[N(SiMe$_3$)$_2$]$_2$ via syringe at −78°C. The mixture was stirred for 30 min, allowed to warm to room temperature and filtered. The colourless residue was washed with petroleum ether (3 × 30 cm^3) and dried *in vacuo* to give a pale pink solid, m.p. >220°C (0.64 g, 0.114 mmol, 52%). Found C, 38.3; H, 3.9. Calc. C$_{18}$H$_{22}$Te$_2$Zn: C, 38.7; H, 4.0% IR (Nujol mull, cm^{-1}): 355 m, 311 w, 218 sh, 178 s. ^{1}H NMR (pyridine-d_5): δ 2.12 (s, 6 H, *p*-Me), 2.64 (s, 12 H, *o*-Me), 6.75 (s, 4 H, aryl-H). ^{13}C NMR: δ 20.76 (*p*-Me), 32.60 (*o*-Me), 113.87 (*m*-C), 126.63 (*ipso*-C), 134.82 (*p*-C), 145.64 (*o*-C).

Synthesis of Lewis-base adducts

The synthesis of zinc complexes is exemplified below for Zn(TeR^1)$_2$(PMe$_3$) (**2**).

Zn(TeR^1)$_2$(PMe$_3$) (**2**). To a suspension of 0.330 g **1** (0.59 mmol) in 10 cm^3 petroleum ether was added via syringe 0.040 g (0.53 mmol) PMe$_3$. The starting material dissolved within a few seconds, followed by the formation of a colourless precipitate. Stirring was continued for 2 h. The product was washed with petroleum ether (3 × 20 cm^3) and dried *in vacuo*. Prolonged exposure to vacuum causes partial loss of phosphine ligand. Yield 0.188 g (0.296 mmol, 56%), m.p. 116–118°C. Found C, 38.9; H, 4.2. Calc. C$_{21}$H$_{31}$PTe$_2$Zn: C, 39.7; H, 4.9% IR (Nujol mull, cm^{-1}): 339 w, 318 w, 215 sh, 196 s. ^{1}H NMR (CDCl$_3$): δ 1.07 (d, 9 H, PMe$_3$, J_{PH} = 4.3 Hz), 2.22 (s, 6 H, *p*-Me), 2.56 (s, 12 H, *o*-Me), 6.82 (s, 4 H, aryl-H). ^{13}C NMR: δ 12.26 (d, PMe$_3$, J_{PC} = 17 Hz), 20.72 (*p*-Me), 32.20 (*o*-Me), 126.43 (*ipso*-C), 135.22 (*p*-C), 145.18 (*o*-C). ^{31}P NMR: δ −47.3.

Zn(TeR^1)$_2$(PMe$_3$)$_2$ (**3**). Yield 49%, m.p. 106–108°C. Found C, 38.5; H, 5.1. Calc. C$_{24}$H$_{40}$P$_2$Te$_2$Zn: C, 40.5; H, 5.7% ^{1}H NMR (CDCl$_3$): δ 1.10 (d, 18 H, PMe$_3$, J_{PH} = 4.3 Hz), 2.20 (s, 6 H, *p*-Me), 2.57 (s, 12 H, *o*-Me), 6.82 (s, 4 H, aryl-H). ^{13}C NMR: δ 12.45 (d, PMe$_3$, J_{PC} = 15.8 Hz), 20.72 (*p*-Me), 32.46 (*o*-Me), 112.83 (*m*-C), 126.09 (*ipso*-C), 134.82 (*p*-C), 145.50 (*o*-C). ^{31}P NMR: δ −47.8.

Zn(TeR^1)$_2$(dmpe) (**4**). Yield 59%. Found C, 38.9; H, 5.2. Calc. C$_{24}$H$_{44}$P$_2$Te$_2$Zn: C, 40.7; H, 5.4%. IR (Nujol mull, cm^{-1}): 315 w, 261 w, 202 sh, 190 s. ^{1}H NMR (pyridine-d_5): δ 0.95 (d, 12 H, PMe$_2$, J_{PH} = 1.3 Hz), 1.42 (t, 4 H, J_{PH} = 3.8 Hz, C$_2$H$_4$), 2.12 (s, 6 H, *p*-Me), 2.65 (s, 12 H, *o*-Me), 6.75 (s, 4 H, aryl-H). ^{13}C NMR: δ 13.84 (PMe$_2$), 20.72 (*p*-Me), 27.69 (C$_2$H$_4$), 32.58 (*o*-Me), 122.71 (*m*-C), 126.61 (*ipso*-C), 134.79 (*p*-C), 145.62 (*o*-C).

Zn(TeR^1)$_2$(imid)$_2$ (**5**). Yield 62%, m.p. 152–154°C. Found C, 42.1; H, 4.6; N, 7.7. Calc. C$_{26}$H$_{34}$N$_4$Te$_2$Zn: C, 43.2; H, 4.7; N, 7.8%. IR (Nujol mull, cm^{-1}): 353 m, 314 w, 218 sh, 200 s. ^{1}H NMR (CDCl$_3$): δ 2.15 (s, 6 H, *p*-Me), 2.38 (s, 12 H, *o*-Me), 3.50 (s, 6 H, N-Me), 6.70 (s, 4 H, aryl-H), 6.75–7.00 (m, 6 H, imid-H). ^{13}C NMR: δ 20.76 (*p*-Me), 32.24 (*o*-Me), 33.91 (N-Me), 120.25 (*m*-C), 125.66 (*ipso*-C), 134.09 (*p*-C, imid), 138.63 (imid), 145.82 (*o*-C).

Zn(TeR^1)$_2$(C$_5$H$_5$N)$_2$ (**6**). Yield 57%, m.p. 114–116°C (dec.). Found C, 46.8; H, 4.2; N, 3.5. Calc. C$_{28}$H$_{32}$N$_2$Te$_2$Zn: C, 46.9; H, 4.5; N, 3.9%. ^{1}H NMR (pyridine-d_5): δ 2.14 (s, 6 H, *p*-Me), 2.65 (s, 12 H, *o*-Me), 6.76 (s, 4 H, aryl-H), 7.21, 7.58, 8.74 (py).

X-ray crystallography

Crystals of **6** were grown from pyridine at 10°C as colourless blocks. X-ray measurements were made using a crystal of dimensions 0.20 × 0.25 × 0.40 mm mounted in a sealed capillary tube under argon.

Crystal data. C$_{28}$H$_{32}$N$_2$Te$_2$Zn, M = 717.15, monoclinic, space group $C2/c$, a = 13.913(4) Å, b = 13.848(4) Å, c = 15.794(5) Å, β = 110.23(2)°, V = 2855.3(15) Å^3, Z = 4, ρ = 1.668 g cm^{-3}, absorption coefficient (Mo-K_α radiation) = 2.907 mm^{-1}, F(000) = 1392.

Data collection. Data were collected at room temperature on a Siemens R3m/V diffractometer using Mo-K_α radiation (λ = 0.71073 Å) and a graphite monochromator with ω scans in the range $2 \leqslant 2\theta \leqslant 50°$ with variable scan speeds (1.5 to 14.65° min^{-1} in ω) from $0 \leqslant h \leqslant 16$, $0 \leqslant k \leqslant 16$, $-18 < l < 17$. Of the 5526 reflections collected, 2540 were independent with 1964 considered as observed with $|F| > |4.0\ \sigma(F)|$. A semi-empirical absorption correction based on azimuthal psi scans was applied (max/min transmission = 0.0741/0.0374).

Structure solution and refinement. The structure was solved by direct methods of phase determination using the SHELXTL(Plus) package and refined using successive cycles of full-matrix least squares to final R values of $R = 3.63\%$ and $wR = 6.83\%$ ($w^{-1} = \sigma^2(F) + 0.0060\ F^2$). Residual electron density was $+0.55$ and -0.37 eÅ^{-3}. Hydrogens were allowed to ride on their parent atoms with fixed isotropic U.

Thermolysis studies. Thermolysis of dry solids was carried out by placing *ca* 350 mg samples in a pyrex or quartz tube. About three-quarters of the evacuated tube was inserted in a tube furnace, leaving one end at room temperature while the end containing the sample was heated to the temperature given in Table 2. Volatile material condensed in the cool end where it was collected for analysis by NMR and mass spectrometry. The solid thermolysis residue, typically 96–98% of the calculated weight, was ground to a fine powder prior to X-ray diffraction.

Thermolysis reactions in paraffin oil were done by heating 0.583 g of **1** under nitrogen in 30 cm^3 of the oil at 270°C for 6 h. The mixture was centrifuged and the fine powdery precipitate was collected, washed with petroleum ether and dried *in vacuo* prior to X-ray measurements. Annealing of a sample in a quartz tube at 700°C for 3 h under argon led to partial decomposition of ZnTe into zinc and tellurium which sublimed to the cooler part of the tube. The residue consisted of ZnTe with much improved crystallinity.

The thermolysis of **1** (0.563 mg) in 4-ethylpyridine (20 cm^3) proceeded more slowly, with darkening of the solution, but initially without precipitation. UV measurements were taken after 5, 20, 45 min and 6 h which showed that decomposition was progressing, as indicated by the formation of $R^1{}_2Te$ ($\lambda_{max} = 388$ nm) and $R^1{}_2Te_2$ ($\lambda_{max} = 435$ nm), though growth of the ZnTe particles was considerably slower than in less strongly coordinating solvents. ZnTe was isolated as a brown powder after washing with petroleum ether (190.8 mg, 98%).

The thermolysis of **2** (29.4 mg) in 4-ethylpyridine (10 cm^3) was monitored by UV–vis spectroscopy after 5, 30, 60, 120 min, 2 and 66 h. Although the spectra gave indications of particle formation, attempts to isolate a solid product using an ultracentrifuge at 35,000 rpm were unsuccessful. A small quantity of solid was obtained after 90 h.

Additional material available from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 IEZ comprise full lists of bond lengths and angles, atomic coordinates, anisotropic displacement coefficients and H-atom coordinates.

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